

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2003-040687

(43)Date of publication of application : 13.02.2003

(51)Int.Cl.

C04B 38/00  
B01D 39/20  
B28B 3/26  
C04B 35/195  
F01N 3/02

(21)Application number : 2001-187245

(71)Applicant : NGK INSULATORS LTD

(22)Date of filing : 20.06.2001

(72)Inventor : KUMAZAWA KAZUHIKO  
NOGUCHI YASUSHI  
NISHI HIDEAKI  
SUENOBU HIROYUKI

(30)Priority

Priority number : 2000199801  
2001157114Priority date : 30.06.2000  
25.05.2001

Priority country : JP

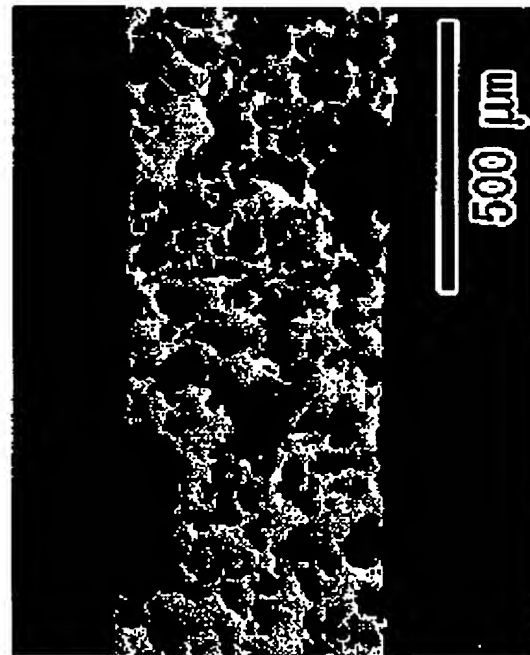
JP

## (54) HONEYCOMB STRUCTURED CERAMIC COMPACT AND ITS MANUFACTURING METHOD

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a honeycomb structured ceramic compact capable of attaining low pressure loss and high capturing efficiency, and to provide its manufacturing method.

SOLUTION: The chemical composition of this honeycomb structured ceramic compact is composed of 42-56 wt.% SiO<sub>2</sub>, 30-45 wt.% Al<sub>2</sub>O<sub>3</sub>, and 12-16 wt.% MgO, the main ingredient of crystal phase of which is cordierite. The porosity is 55-65%, mean fine pore diameter is 15-30  $\mu$ m, and total area of the fine pores exposing on a partition which is constituting the honeycomb ceramic structured, to total area of partition surface is  $\geq$ 35%. The honeycomb structured compact is manufactured by kneading the cordierite material with 15-25 wt.% graphite as a void forming agent and 5-15 wt.% synthetic resin, forming into honeycomb shape, then drying and firing.



## LEGAL STATUS

[Date of request for examination]

27.01.2003

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application]

BEST AVAILABLE COPY

\* NOTICES \*

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

---

CLAIMS

---

[Claim(s)]

[Claim 1] Chemical composition is SiO<sub>2</sub>. 42 - 56 % of the weight, aluminum 2O<sub>3</sub> 30 - 45 % of the weight, MgO The honeycomb ceramic structure to which the gross area of the pore exposed to the septum front face which it is the honeycomb ceramic structure to which it consists of 12 - 16 % of the weight, and a crystal phase uses cordierite as a principal component, and porosity is 55 - 65%, and average pore size is 15-30 micrometers, constitutes this honeycomb ceramic structure be characterized by being 35% or more of a gross area on this front face of a septum.

[Claim 2] The honeycomb ceramic structure according to claim 1 whose gross area of the pore exposed to the septum front face is 40% or more of a gross area on this front face of a septum.

[Claim 3] The honeycomb ceramic structure according to claim 1 or 2 whose average pore size is 15-25 micrometers.

[Claim 4] The honeycomb ceramic structure given in any 1 term of claims 1-3 whose septum thickness is 300 micrometers or less.

[Claim 5] The honeycomb ceramic structure given in any 1 term of claims 1-4 1.5-6 micrometers of whose permeability coefficients are 2.

[Claim 6] The honeycomb ceramic structure given in any 1 term of claims 1-5 whose coefficients of thermal expansion between 40-800 degrees C are less than [  $0.5 \times 10^{-6} / \text{degree C}$  ].

[Claim 7] The honeycomb ceramic structure given in any 1 term of claims 1-6 used as a diesel particulate filter.

[Claim 8] After adding five to 15% of the weight, kneading synthetic resin and fabricating graphite in the shape of a honeycomb subsequently to 15 to 25% of the weight as an ostomy agent in a cordierite-ized raw material, by drying and calcinating Chemical composition is SiO<sub>2</sub>. 42 - 56 % of the weight, aluminum 2O<sub>3</sub> 30 - 45 % of the weight, MgO Consist of 12 - 16 % of the weight, and a crystal phase uses cordierite as a principal component. The gross area of the pore exposed to the septum front face which porosity is 55 - 65%, and average pore size is 15-30 micrometers, and constitutes this honeycomb ceramic structure The manufacture approach of the honeycomb ceramic structure characterized by manufacturing the honeycomb ceramic structure which is 35% or more of a gross area on this front face of a septum.

[Claim 9] The manufacture approach of the honeycomb ceramic structure according to claim 8 that synthetic resin is any one of polyethylene terephthalate (PET), a polymethyl methacrylate (PMMA), bridge formation polystyrene, and the phenol resin, or such combination.

[Claim 10] The manufacture approach of the honeycomb ceramic structure according to claim 8 or 9 that the mean particle diameter of the talc raw material in a cordierite-ized raw material is [ the mean particle diameter of a silica raw material ] 60 micrometers or less in 50 micrometers or less.

---

[Translation done.]

**\* NOTICES \***

**JPO and NCIPi are not responsible for any damages caused by the use of this translation.**

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

---

**DETAILED DESCRIPTION**

---

**[Detailed Description of the Invention]**

**[0001]**

**[Field of the Invention]** High collection efficiency is acquired for example, by low voltage force loss, and this invention relates to the honeycomb ceramic structure which can be suitably used as a diesel particulate filter (DPF), and its manufacture approach.

**[0002]**

**[Description of the Prior Art]** In recent years, the diesel particulate filter (DPF) which carries out uptake of the particulate discharged by the Diesel engine is capturing the spotlight, and DPF from which high collection efficiency is acquired by low voltage force loss is demanded. As DPF, the honeycomb structure object made from cordierite is used from the former, and amelioration of the porosity of a honeycomb structure object, pore distribution, etc. has been performed from the former in order to acquire high collection efficiency by the above low voltage force loss.

**[0003]** The honeycomb structure object which specified the pore distribution in a septum front face in JP,9-77573,A while enlarging porosity and average pore size is indicated, and while making septum thickness thin below predetermined, the honeycomb structure object which enlarged porosity is indicated by JP,11-333293,A.

**[0004]** Moreover, manufacturing the honeycomb structure object of high porosity by using the thing of coarse grain as both particles of a talc component and a silica component among cordierite-ized raw materials more than predetermined is indicated, and while enlarging porosity, the honeycomb structure object which specified pore distribution and surface roughness is indicated by the patent No. 2726616 official report at JP,7-38930,B.

**[0005]** In the above-mentioned conventional technique, although the cordierite-ized raw material was made into the big and rough particle or adding graphite, wood flour, a foaming agent, etc. as an ostomy agent was performed in order to gather porosity, sufficient effectiveness was not able to be acquired.

**[0006]** That is, when a cordierite-ized raw material was used as coarse grain, it was difficult for a cordierite-ized reaction not to fully advance but to attain low-fever expansion. Moreover, since it became difficult to carry out uniform desiccation and 800-1000-degree C firing time was further lengthened in the baking process by dielectric drying or microwave desiccation when graphite was used as an ostomy agent, the dielectric constant of the Plastic solid which added graphite fell and the addition increased, there were problems, like there is the need of controlling rapid combustion of graphite.

**[0007]** Moreover, while addition of a lot of water was needed and the effectiveness of a desiccation process worsened in order to make a plastic matter into a predetermined degree of hardness in a kneading process when starch and wood flour were used as an ostomy agent, also in the baking process, starch and wood flour were difficult to burn rapidly among 200-400 degrees C, and to prevent a lifting and a baking crack for big generation of heat. Thus, in the conventional technique, it was very difficult to make porosity larger than predetermined.

**[0008]**

**[Problem(s) to be Solved by the Invention]** Then, as a result of inquiring wholeheartedly in view of the above-mentioned conventional technical problem, while gathering the porosity of a honeycomb structure object more than predetermined, this invention person A header and this invention are reached [ that exhaust gas actually contacts, and it becomes pressure loss very low when total of the pore area exposed to the septum front face is carried out to more than predetermined paying attention to the pore area which exists in the septum front face through which exhaust gas passes, and high collection efficiency can be attained, and ].

[0009]

[Means for Solving the Problem] That is, according to this invention, chemical composition is SiO<sub>2</sub> 42 - 56 % of the weight, aluminum 2O<sub>3</sub> 30 - 45 % of the weight, MgO It is the honeycomb ceramic structure to which it consists of 12 - 16 % of the weight, and a crystal phase uses cordierite as a principal component. Porosity is 55 - 65%, average pore size is 15-30 micrometers, and the honeycomb ceramic structure to which the gross area of the pore exposed to the septum front face which constitutes the honeycomb ceramic structure is characterized by being 35% or more of a gross area on the front face of a septum is offered.

[0010] In the honeycomb ceramic structure of this invention, it is desirable that the gross area of the pore exposed to the septum front face is 40% or more of a gross area on the front face of a septum, and average pore size is 15-25 micrometers. Furthermore, it is desirable that septum thickness is 300 micrometers or less. Moreover, it is desirable that 1.5-6 micrometers of permeability coefficients are 2. Moreover, as for the honeycomb ceramic structure of this invention, it is desirable for the coefficient of thermal expansion between 40-800 degrees C to be less than [  $0.5 \times 10^{-6} / \text{degree C}$  ].

[0011] The honeycomb ceramic structure of this invention can be preferably used as a diesel particulate filter (DPF) which carries out uptake of the particulate discharged by the Diesel engine.

[0012] After according to this invention adding five to 15% of the weight, kneading synthetic resin and fabricating graphite in the shape of a honeycomb subsequently to 15 to 25% of the weight as an ostomy agent in a cordierite-ized raw material, moreover, by drying and calcinating Chemical composition is SiO<sub>2</sub> 42 - 56 % of the weight, aluminum 2O<sub>3</sub> 30 - 45 % of the weight, MgO Consist of 12 - 16 % of the weight, and a crystal phase uses cordierite as a principal component. The gross area of the pore exposed to the septum front face which porosity is 55 - 65%, and average pore size is 15-30 micrometers, and constitutes this honeycomb ceramic structure The manufacture approach of the honeycomb ceramic structure characterized by manufacturing the honeycomb ceramic structure which is 35% or more of a gross area on this front face of a septum is offered.

[0013] In the above, as synthetic resin, it is desirable that it is any one of polyethylene terephthalate (PET), a polymethyl methacrylate (PMMA), and the phenol resin or such combination, and the mean particle diameter of the talc raw material in a cordierite-ized raw material is 50 micrometers or less, and it is desirable that the mean particle diameter of a silica raw material is 60 micrometers or less.

[0014]

[Embodiment of the Invention] Hereafter, this invention is further explained to a detail. For the honeycomb ceramic structure of this invention, chemical composition is SiO<sub>2</sub> 42 - 56 % of the weight, aluminum 2O<sub>3</sub> 30 - 45 % of the weight, MgO It consists of 12 - 16 % of the weight, and a crystal phase uses cordierite as a principal component, the porosity is 55 - 65%, and average pore size is 15-30 micrometers, and the gross area of the pore exposed to the septum front face which constitutes the honeycomb ceramic structure is 35% or more of a gross area on the front face of a septum.

[0015] In the honeycomb ceramic structure of this invention, the range of the porosity is 55 - 65%. When porosity is less than 55%, the pressure loss of exhaust gas goes up, and preferably, on the other hand, if porosity exceeds 65%, the mechanical strength of a honeycomb structure object falls remarkably, and cannot bear real use.

[0016] Moreover, in this honeycomb ceramic structure, it is desirable that average pore size is 15-30 micrometers, and it is 15-25 micrometers. Although pressure loss is low and it is good if it becomes [ pressure loss ] high and is not desirable although collection efficiency rises and average pore size exceeds 30 micrometers when average pore size is less than 15 micrometers, the probability for uptake of the particle in exhaust gas not to be carried out through an atmospheric-air hole becomes high. Especially, when the wall thickness of the septum of the honeycomb ceramic structure is 300 micrometers or less, decline in collection efficiency becomes remarkable. Moreover, although early pressure loss has low average pore size when porosity is less than 55% exceeding 30 micrometers, there is an inclination for pressure loss to go up rapidly as a time progresses. The particle in exhaust gas becomes easy to deposit this on the interior of a septum through an atmospheric-air hole, and it is presumed since possibility of burning and remaining inside a septum at the time of combustion playback becomes high. Furthermore, it burns, remains and deposits on a septum front face inside a septum similarly in the thing of the continuation playback type which supported the oxidation catalyst, and it is presumed that pressure loss is raised. As for this to average pore size, it is more desirable that it is the range of 15-25 micrometers.

[0017] Moreover, in this invention, the gross area of the pore exposed to the septum front face which constitutes the honeycomb ceramic structure is 35% or more of a gross area on the front face of a septum. Thus, by having made larger than predetermined the gross area of the pore exposed to the septum front face,

to exhaust gas, it is very low pressure loss, and high collection efficiency can be attained. In addition, 40% or more of the gross area on the front face of a septum of the gross area of the pore exposed to the septum front face is desirable, and it is desirable that it is 60% or less.

[0018] Moreover, in the honeycomb structure object of this invention, 1.5-6 micrometers of permeability coefficients can be set to 2. With the honeycomb structure object which makes a permeability coefficient this range, low pressure loss can attain high collection efficiency to exhaust gas. Here, in this specification, a permeability coefficient means the numeric value calculated based on a degree type.

[0019]

[Equation 1]

$$C = \frac{8FTV}{\pi D^2 (P^2 - 13.839^2) / 13.839 \times 68947.6} \times 10^8$$

[0020] the inside of "type, and C -- in the sample path length (cm) and V, gas viscosity (dynes-s/cm<sup>2</sup>) and D show a sample diameter (cm), and, as for a permeability coefficient (micrometer<sup>2</sup>) and F, P shows [ a gas flow rate (cm<sup>3</sup>/s) and T ] gas pressure (PSI), respectively. Moreover, the numeric value shown in a formula is 13.839(PSI) = 1 (atm), and is 68947.6(dynes/cm<sup>2</sup>) = 1 (PSI). "

[0021] The coefficient of thermal expansion between 40-800 degrees C can make the honeycomb ceramic structure of this invention less than [ 0.5x10<sup>-6</sup>/degree C ]. In having such a coefficient of thermal expansion, even if the extremely excellent thermal shock resistance is shown and a rapid temperature change occurs repeatedly, there is almost no possibility of damaging. Moreover, since collection efficiency is high, the wall thickness of a septum can apply the honeycomb ceramic structure of this invention to the honeycomb structure object of the thin wall of [ having described above ] 300 micrometers or less preferably.

[0022] Therefore, the honeycomb ceramic structure of this invention which has the above-mentioned configuration can be applied very preferably as a diesel particulate filter (DPF) which carries out uptake of the particulate discharged by the Diesel engine.

[0023] Next, the manufacture approach of the honeycomb ceramic structure concerning this invention is explained.

[0024] First, talc, a kaolin, a temporary-quenching kaolin, an alumina, an aluminum hydroxide, The chemical composition out of a silica is SiO<sub>2</sub>. 42 - 56 % of the weight, 2O<sub>3</sub>30 - 45 % of the weight of aluminum, MgO In the cordierite-ized raw material prepared by the predetermined rate so that it might go into 12 - 16% of the weight of the range Synthetic resin, such as 15 - 25 % of the weight and PET and PMMA, and phenol resin, is added for graphite five to 15% of the weight as an ostomy agent, after specified quantity addition and water are added suitably, methyl cellulose and a surfactant are kneaded, and it considers as a plastic matter. Subsequently, after carrying out extrusion molding of this plastic matter to honeycomb structure after a vacuum deairing and drying by dielectric drying or microwave desiccation, and the hot-air-drying method, the honeycomb ceramic structure concerning this invention can be manufactured according to a series of processes of calcinating a maximum temperature among 1400-1435 degrees C.

[0025] Moreover, the alternate end-face closure in the honeycomb ceramic structure is carried out after desiccation or baking, and is performed by calcinating a honeycomb structure object again etc.

[0026] In the manufacture approach of this invention, while adding the graphite used for a cordierite-ized raw material as an ostomy agent 15 to 25% of the weight, calorific value at the time of combustion is characterized by adding synthetic resin, such as comparatively small PET, PMMA, or phenol resin, five to 15% of the weight, and \*\* of it which mass-produces cheaply a cordierite honeycomb structure object with 55% or more of porosity became possible by this.

[0027] When the addition of the graphite to a cordierite-ized raw material exceeded 25 % of the weight, while it became difficult to carry out uniform desiccation by dielectric drying and microwave desiccation, at the baking process, firing time which is 800-1000 degrees C at which graphite burns needed to be lengthened, and rapid combustion of graphite needed to be controlled. When the programming rate of the combustion zone of this graphite is too quick, graphite burns rapidly, big temperature distribution arise and the danger that a crack will occur is in the honeycomb structure. Moreover, when graphite burned and remains, it has a bad influence on the cordierite-ized reaction 1200 degrees C or more in an elevated temperature, and there is a danger that thermal expansion will become high. For this reason, the need has that it is 25 or less % of the weight, and the addition of graphite is 20 or less % of the weight more preferably, when industrial mass production is considered. If an example is taken in ostomy nature and calorific value, 15 % of the weight or more is required for the amount of minimums of addition of graphite.

[0028] In this invention, it becomes possible by carrying out specified quantity addition of the synthetic

resin with the comparatively small calorific value at the time of combustion to manufacture the honeycomb structure object of the big porosity of 55% or more at this graphite.

[0029] Moreover, in order to enlarge the gross area of the pore exposed to the honeycomb structure object septum front face like the honeycomb ceramic structure of this invention, while enlarging porosity, it is necessary to control the pore formed in process of [ silica / talc and ] a cordierite-ized reaction. Although average pore size can be enlarged when a talc raw material and a silica raw material are used as coarse grain, the formed pore does not necessarily appear in a septum front face, but remains for forming big and rough pore in the interior of a septum. This is because big and rough particles tend to gather centering on a septum at the time of extrusion molding.

[0030] Then, in this invention, the rate to the gross area on the front face of a septum of the gross area of the pore which became possible [ forming pore in a septum front face effectively ], consequently was exposed to the septum front face of a honeycomb structure object can be enlarged to 35% or more by controlling the mean particle diameter of 50 micrometers or less and a silica raw material for the mean particle diameter of a talc raw material important for pore formation to 60 micrometers or less. In addition, the mean particle diameter of the range of 20-50 micrometers and a silica raw material has [ the mean particle diameter of a talc raw material ] the more desirable range of 20-60 micrometers.

[0031]

[Example] Hereafter, although this invention is further explained to a detail based on an example, this invention is not limited to these examples.

(Examples 1-11, examples 1-16 of a comparison) It prepared, respectively in proportion which shows the cordierite-ized raw material shown in Table 1, and an ostomy agent in Table 2, fatty-acid soap was respectively added for methyl cellulose and hydroxypropoxyl methyl cellulose 0.5% of the weight as a surfactant 2% of the weight, water was added suitably, and it considered as the plastic matter. Subsequently, using this plastic matter, as shown in Table 3, the cellular structure carried out extrusion molding of various kinds of honeycomb structure objects of wall thickness:300micrometer, number of cels:31 cel / cm<sup>2</sup> or wall thickness:430micrometer, and number of cels:16 cel / cm<sup>2</sup> by phi 150mmx150mm (die length), and moisture was removed by dielectric drying and hot air drying. Then, it calcinated on the conditions of maximum-temperature holding-time 8 hours by 1415 degrees C of maximum temperatures, and after carrying out eye closure from a slurry-like cordierite-ized raw material so that it may become alternate by turns about an end face, it calcinated again at 1420 degrees C of maximum temperatures, and the evaluation sample of various kinds of honeycomb ceramic structures was created. The physical properties and evaluation result of the honeycomb ceramic structure which were obtained are shown in Table 3.

[0032]

[Table 1]

原料名		平均粒径	化学分析(%)						
		( $\mu\text{m}$ )	IgLoss	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	MgO	CaO+Na <sub>2</sub> O+K <sub>2</sub> O
主原料	タルクA	25	5.5	62	0.15	1.75	0.005	31	0.15
	タルクB	45	5	63	0.1	0.02	0	31.5	0.5
	タルクC	25	8.5	59.5	0.7	2.5	0.02	30.5	0.02
	タルクD	35	4.8	62.5	0.2	0.4	0.01	31.7	0.1
	タルクE	55	5	63	0.1	0.02	0	31.5	0.5
	焼タルク	25	0	66.3	0.1	0.0	0.0	33.2	0.5
	カオリンA	9	14	45.5	39	0.2	0.7	0.01	0.09
	カオリンB	4	13.5	46	39	0.4	0.8	0.02	0.1
	カオリンC	5	14	45.5	39	0.3	0.7	0.01	0.1
	アルミナ	8	0.05	0.02	99.5	0.02	0	0	0.2
	水アルA	1	34	0	65.5	0	0	0	0.35
	水アルB	2	34	0.05	65.5	0	0	0	0.3
	シリカA	20	0.1	99.8	0.02	0.02	0	0	0.02
	シリカB	110	0.1	99.7	0.1	0	0	0	0.01
造孔剤	シリカC	40	0.1	99.8	0.1	0.02	0	0	0.01
	シリカD	50	0.1	99.8	0.02	0.02	0	0	0.02
	溶融シリカ	42	0.1	99.8	0.1	0.02	0	0	0.01
	グラファイト	40	99.5			0.2			
	PET	60	99.9						
	PMMA	60	99.9						
	フェノール樹脂	60	99.8						
	コーンスターチ	60	99.8						
	クレミ粉	150	99.8						

[0033]

[Table 2]

バッチNo.	コーゼライト化原料割合 (wt%)										原料平均粒径(μm)	
	タルク	焼タルク	カオリン	焼カオリ ン	アルミナ	水酸化 アルミニウム	シリカ	熔融シリカ	造孔剤(wt%)	タルク原料	シリカ原料	
1	B: 28	10	B: 14	10	11.5	A: 16.5	C: 10	0	ｸﾞﾗﾌｱｲﾄ:20	43	40	
2	AB(1:1) 41	0	B: 16	0	15	B: 16	A: 12	0	ｸﾞﾗﾌｱｲﾄ:20	35	20	
3	A: 41	0	B: 16	0	15	B: 16	A: 12	0	ｸﾞﾗﾌｱｲﾄ:20	25	20	
4	AB(1:1) 41	0	C: 16	0	15	B: 16	D: 12	0	ｸﾞﾗﾌｱｲﾄ:20	35	50	
5	A: 41	0	B: 16	0	15	B: 16	B:D(5:7) 12	0	ｸﾞﾗﾌｱｲﾄ:20	25	75	
6-1	A: 40	0	A: 17	0	15	A: 16	0	12	ｸﾞﾗﾌｱｲﾄ:30	25	42	
6-2	A: 40	0	A: 17	0	15	A: 16	0	12	ｸﾞﾗﾌｱｲﾄ:20+ｺﾝｽﾀｰｽﾞ:10	25	42	
6-3	A: 40	0	A: 17	0	15	A: 16	0	12	ｸﾞﾗﾌｱｲﾄ:20+ｸﾙﾐﾝ粉:10	25	42	
6	A: 40	0	A: 17	0	15	A: 16	0	12	ｸﾞﾗﾌｱｲﾄ:15+PET:15	25	42	
7	C: 39	0	B: 15	0	15	B: 17	B: 3	11	ｸﾞﾗﾌｱｲﾄ:15+PET:15	25	57	
8	B: 39	0	B: 14	0	15	B: 17	B: 4	11	ｸﾞﾗﾌｱｲﾄ:15+PET:15	50	60	
9	A: 39	0	B: 15	0	15	B: 17	B: 3	11	ｸﾞﾗﾌｱｲﾄ:15+PET:15	25	57	
10	D: 39	0	B: 14	0	15	B: 17	B: 4	11	ｸﾞﾗﾌｱｲﾄ:15+PET:15	35	60	
11	BC(1:1) 39	0	B: 14	0	15	B: 17	B: 4	11	ｸﾞﾗﾌｱｲﾄ:15+PET:15	38	60	
12	C: 41	0	B: 16	0	15	A: 16	A: 12	0	ｸﾞﾗﾌｱｲﾄ:20+PET:10	25	20	
13	C: 41	0	B: 16	0	15	A: 16	A: 12	0	ｸﾞﾗﾌｱｲﾄ:20+PMMA:10	25	20	
14	C: 41	0	B: 16	0	15	A: 16	A: 12	0	ｸﾞﾗﾌｱｲﾄ:20+ﾌｪｰﾙｼﾝ:10	25	20	
15	C: 40	0	B: 17	0	15	A: 16	0	12	ｸﾞﾗﾌｱｲﾄ:20+PET:10	25	42	
16	C: 41	0	B: 16	0	15	A: 16	A: 12	0	ｸﾞﾗﾌｱｲﾄ:20+PET:10	25	20	
17	E: 41	0	B: 16	0	15	A: 16	A: 12	0	ｸﾞﾗﾌｱｲﾄ:20+PET:10	55	20	
18	C: 41	0	B: 16	0	15	A: 16	B:D(1:1)12	0	ｸﾞﾗﾌｱｲﾄ:20+PET:10	25	80	
19	E: 41	0	B: 16	0	15	A: 16	B:D(1:1)12	0	ｸﾞﾗﾌｱｲﾄ:20+PET:10	55	80	
20	A: 41	0	B: 16	0	15	A: 16	A: 12	0	ｸﾞﾗﾌｱｲﾄ:20	25	20	
21	A: 40	0	B: 20	0	14	A: 16	A: 10	0	ｸﾞﾗﾌｱｲﾄ:20	25	20	

[0034]

[Table 3]



NO.	素地	壁厚(μm)	セル数(セル/cm <sup>2</sup> )	CTE	平均細孔径(μm)	気孔率(%)	面積率(%)	初期圧損(mmHg)	捕集効率(%)	パーミアリティ(μm <sup>2</sup> )
1	ハッチ1	300	31	0.9	25	52	25	80	80	4.1
2	ハッチ2	300	31	0.5	30	50	23	85	85	5.3
3	ハッチ3	300	31	0.3	16	54	30	85	85	1.4
4	ハッチ4	300	31	0.8	30	55	32	80	90	6.1
5	ハッチ5	300	31	0.4	35	50	20	70	80	7.6
6	ハッチ6-1	300	31							
7	ハッチ6-2	300	31							
8	ハッチ6-3	300	31							
1	ハッチ7	300	31	0.4	25	63	45	65	95	4.8
2	ハッチ7	300	31	0.4	28	60	41	60	95	6.0
3	ハッチ7	300	31	0.5	23	62	43	55	90	4.1
4	ハッチ7	300	31	0.3	28	60	43	65	95	5.9
5	ハッチ10	300	31	0.3	23	62	43	60	95	4.0
6	ハッチ11	300	31	0.4	26	62	42	65	90	5.3
7	ハッチ12	300	31	0.3	17	55	35	75	95	1.9
8	ハッチ13	300	31	0.5	15	58	36	70	85	1.5
9	ハッチ14	300	31	0.3	17	57	39	70	85	2.1
10	ハッチ15	300	31	0.3	20	60	40	85	90	3.0
11	ハッチ16	300	31	0.3	20	58	38	85	90	2.9
8	ハッチ17	300	31	0.6	32	56	34	60	85	7.0
10	ハッチ18	300	31	0.7	35	55	33	60	80	8.3
11	ハッチ19	300	31	0.9	38	53	29	50	70	9.6
12	ハッチ20	300	31	0.3	14	54	26	85	95	1.3
13	ハッチ21	300	31	0.2	10	53	22	95	95	1.0
14	ハッチ12	430	16	0.3	17	55	35	95	98	2.0
15	ハッチ13	430	16	0.4	15	56	36	90	98	1.6
16	ハッチ15	430	16	0.3	20	60	40	85	85	2.9

焼成工程でクラック発生し測定不能

[0035] Here, the rate (rate of area) to the gross area on the front face of a septum of the gross area of the pore exposed to the average pore size of the honeycomb ceramic structure, porosity, and a septum front face, a permeability coefficient, the coefficient of thermal expansion (CTE) for 40-800 degrees C, pressure loss, and collection efficiency were measured as follows.

[0036] Average pore size and porosity are called for from the pore distribution measured by the method of mercury penetration. Porosity was calculated from the total pore volume.

[0037] The rate of area: It asked for the rate of area of the pore exposed to the septum front face in analyzing the photograph on the front face of a septum obtained by SEM observation using an image-analysis processor.



CTE: It was measured by the differential type measuring method by making a quartz into a standard sample.

[0038] Permeability coefficient: What processed some septa from each honeycomb ceramic structure so that drawing and irregularity might be lost is made into a sample, and after putting this sample from the upper and lower sides with a phi20mm sample electrode holder so that there may be no gas leakage, gas was made to flow into a sample with specific gas pressure. Under the present circumstances, based on the formula shown below, the permeability coefficient was searched for about the gas which passed the sample.

[0039]

[Equation 2]

$$C = \frac{8FTV}{\pi D^2 (P^2 - 13.839^2) / 13.839 \times 68947.6} \times 10^8$$

[0040] the inside of "type, and C -- in the sample path length (cm) and V, gas viscosity (dynes-s/cm<sup>2</sup>) and D show a sample diameter (cm), and, as for a permeability coefficient (micrometer<sup>2</sup>) and F, P shows [ a gas flow rate (cm<sup>3</sup>/s) and T ] gas pressure (PSI), respectively. Moreover, the numeric value shown in a formula is 13.839(PSI) = 1 (atm), and is 68947.6(dynes/cm<sup>2</sup>) = 1 (PSI). "

[0041] Pressure loss: The soot was generated with the gas oil gas burner, and it asked from aging of the differential pressure before and behind DPF, depositing on DPF the combustion gas which equips the downstream with DPF and contains 2.4Nm<sup>3</sup>/min of quantities of gas flow, and a soot with a temperature of about 150 degrees C, and making a soot deposit on a sink and DPF.

[0042] Collection efficiency: The soot was generated with the gas oil gas burner, the downstream was equipped with DPF, and the collection effectiveness of DPF was searched for from the ratio of the soot weight in the gas which shunted the combustion gas containing 2.4Nm<sup>3</sup>/min of quantities of gas flow, and a soot with a temperature of about 150 degrees C toward DPF at the rate of a constant ratio from a sink, the upstream of DPF, and each downstream.

[0043] (Consideration) Drawing 1 shows the SEM photograph of the rib cross section of the honeycomb ceramic structure of an example 1, and drawing 2 shows the SEM photograph on the front face of a septum of the honeycomb ceramic structure of an example 1 (film surface). Moreover, drawing 3 shows the SEM photograph of the rib cross section of the honeycomb ceramic structure of the example 5 of a comparison, and drawing 4 shows the SEM photograph on the front face of a septum of the honeycomb ceramic structure of the example 5 of a comparison (film surface). In drawing 2 and two photographs of drawing 4, that which appears in white (it appears yellow with the drawing substitution photograph) is the pore (surface pore) exposed to the septum front face. If the rate of area of surface pore is high, it will lead to the fall of initial pressure loss.

[0044] Drawing 3 and drawing 4 express the microstructure of the honeycomb structure object which is the example 5 of a comparison. The photograph of drawing 3 and drawing 4 shows that very big pores have gathered near the core of a rib with the coarse silica raw material of 75 micrometers of mean diameters in the rib cross section of the example 5 of a comparison. Although it is known that big pore will be formed by using a coarse talc raw material and a silica raw material, a raw material coarse in the case of extrusion molding of a honeycomb does not generate near the core of a rib, and an assembly, consequently big pore generate only near the core of a rib. In the photograph of the film surface of the example 5 of a comparison, the gross area of the pore exposed to the septum front face was only 20%. Although the example 5 of a comparison did not have so high pressure loss, collection efficiency was as bad as 80% under the effect of big pore.

[0045] In the example 1 shown in drawing 1 and drawing 2, in order to raise porosity, PET which is synthetic resin is used as an ostomy agent with graphite. Consequently, porosity became high with 63%. Moreover, when synthetic resin was used, while the porosity of a honeycomb structure object increased, the effectiveness that surface pore increased was checked so that the situation of the upper limit side of the rib cross section of drawing 1 and a lower limit side might show. When image analysis of the photograph of drawing 2 was performed, as shown in Table 3, the rate of area of the surface pore of an example 1 was as high as 45%, consequently 4.8 micrometers of permeability coefficients were 2, it is stopped on 65mmHg(s) and very low level, and initial-pressure loss was the thing of level also with as high collection efficiency as 95%.

[0046] Although starch etc. had been conventionally used as an ostomy agent of a graphite alternative, when it was used abundantly, there was a problem which "cut" generates at a desiccation process or a baking process. Drawing 5 R> 5 is a graph which shows the weight percentage reduction (TG) of the ground

(example 7: batch 12) and the relation of calorific value (DTA) containing the ground (the example 7 of a comparison: batch 6-2), 10 % of the weight (PET) of synthetic resin, and 20 % of the weight of graphite which contained 10 % of the weight (corn starch) of starches, and 20 % of the weight of graphite as an ostomy agent.

[0047] From drawing 5, if starch is used as an ostomy agent, in order that starch may pyrolyze near 300 degrees C - 350 degree C, it will generate heat rapidly (see the dotted line of DTA), and a piece will be generated at a baking process with the thermal stress. However, when PET, PMMA, phenol resin, bridge formation polystyrene, etc. were used as an ostomy agent, the calorific value in the temperature region was stopped low (see the DTA continuous line), and it became clear that there was an advantage that whose a piece is generated in a baking process decreases extremely.

[0048] Drawing 6 is a graph which shows the relation between soot (soot) attachment time amount and pressure loss. The continuous line of drawing 6 is a result about the honeycomb structure object of an example 1, and the broken line of drawing 4 is a result about the honeycomb structure object of the example 5 of a comparison.

[0049] Gas temperature generated with the gas-oil gas burner as soot attachment conditions: It was made to flow into DPF which consists of a honeycomb structure object of an example 1 and the example 5 of a comparison as quantity-of-gas-flow:2.4Nm<sup>3</sup>/min using about 150-degree C thing.

[0050] Even if porosity goes through predetermined time with the honeycomb structure object of an example 1 with the as large rate of area at 63% as 45%, the rise of the pressure loss does not have it, so that the result of drawing 6 may show, but on the other hand, when porosity uses the honeycomb structure object of the example 5 of a comparison with the as small rate of area as 20% at 50%, it turns out that the rise of the pressure loss is large with time amount progress. [ large ]

[0051]

[Effect of the Invention] As explained above, according to this invention, low pressure loss, the honeycomb ceramic structure which can attain high collection efficiency, and its manufacture approach can be offered.

---

[Translation done.]

\* NOTICES \*

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

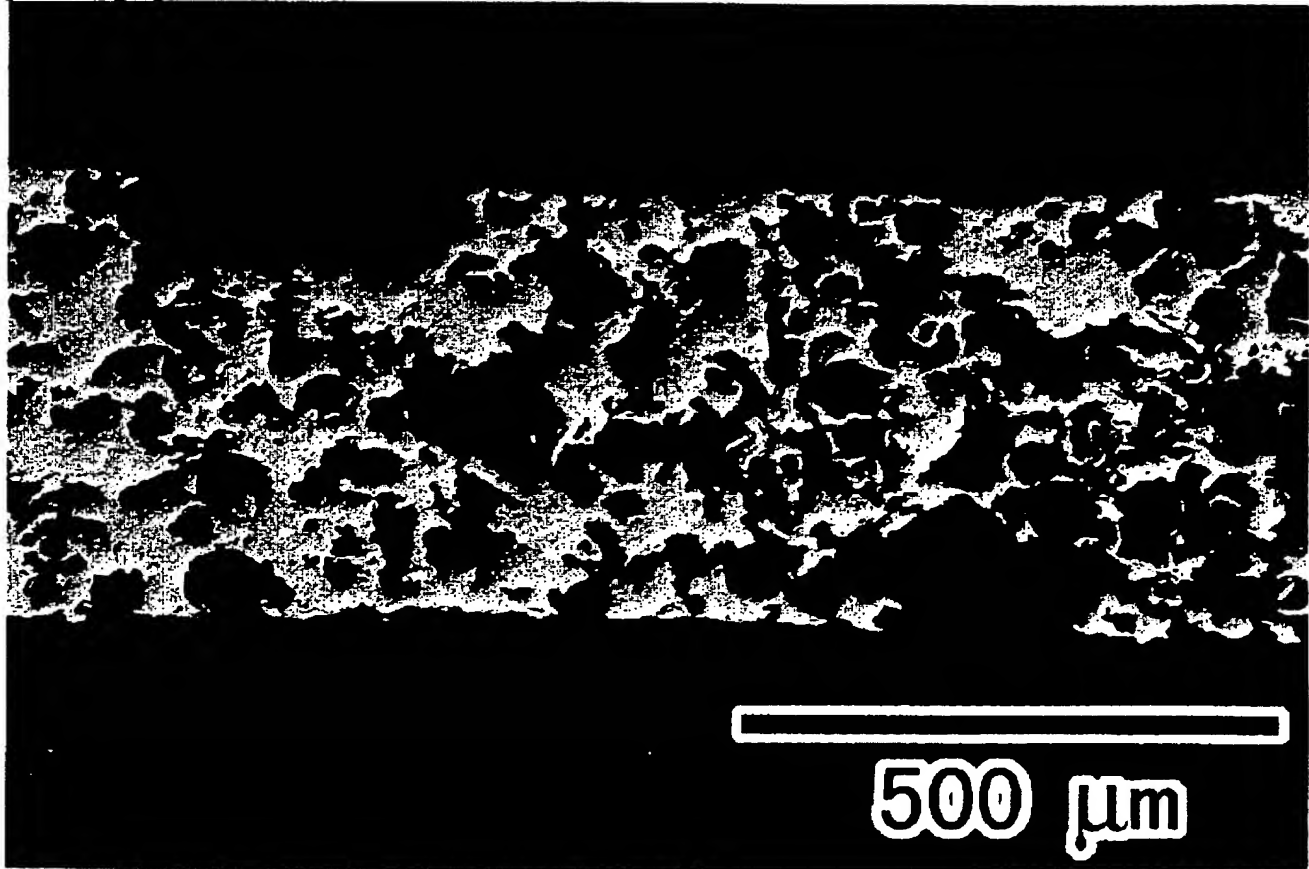
- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

---

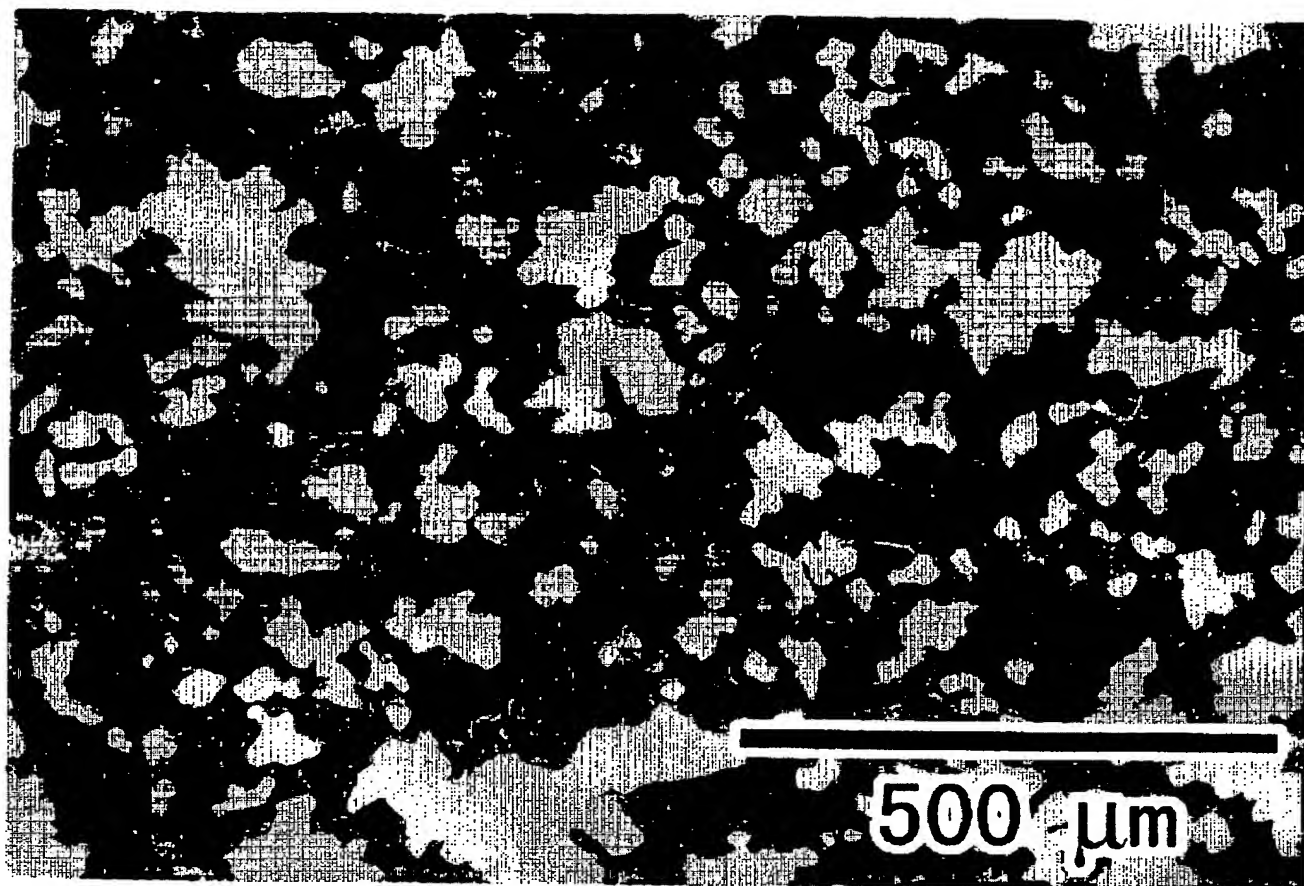
DRAWINGS

---

[Drawing 1]

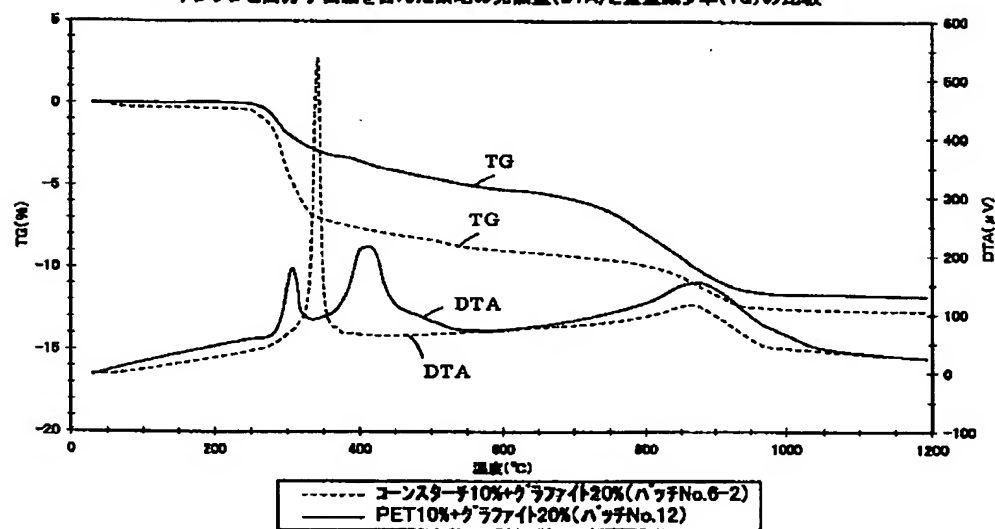


[Drawing 2]

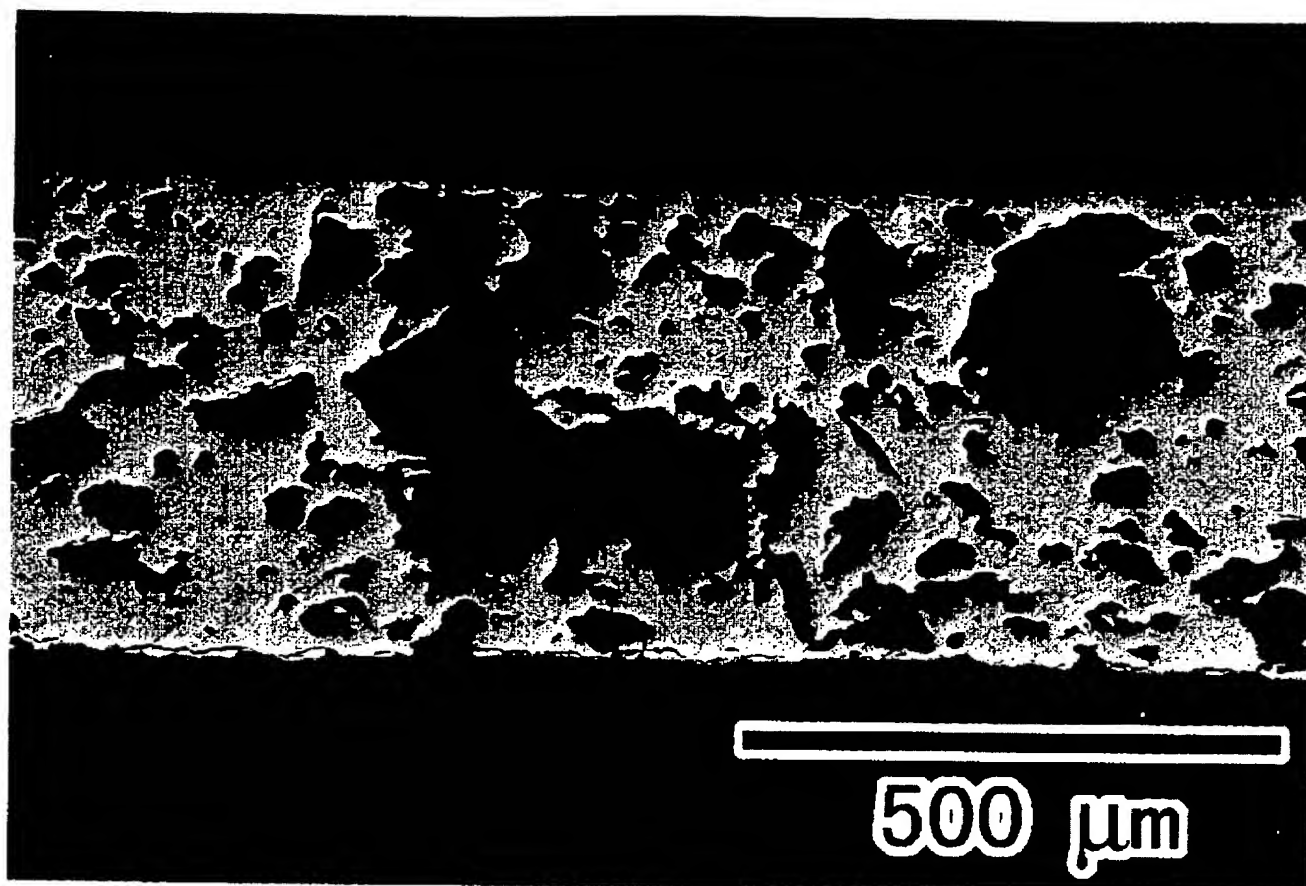


[Drawing 5]

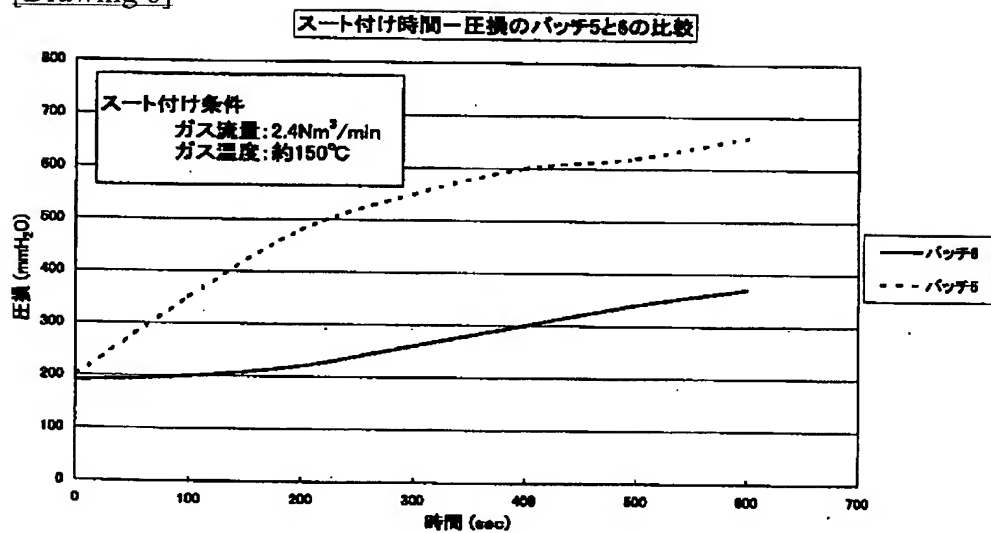
ゼンパンと高分子樹脂を含んだ素地の発熱量(DTA)と重量減少率(TG)の比較



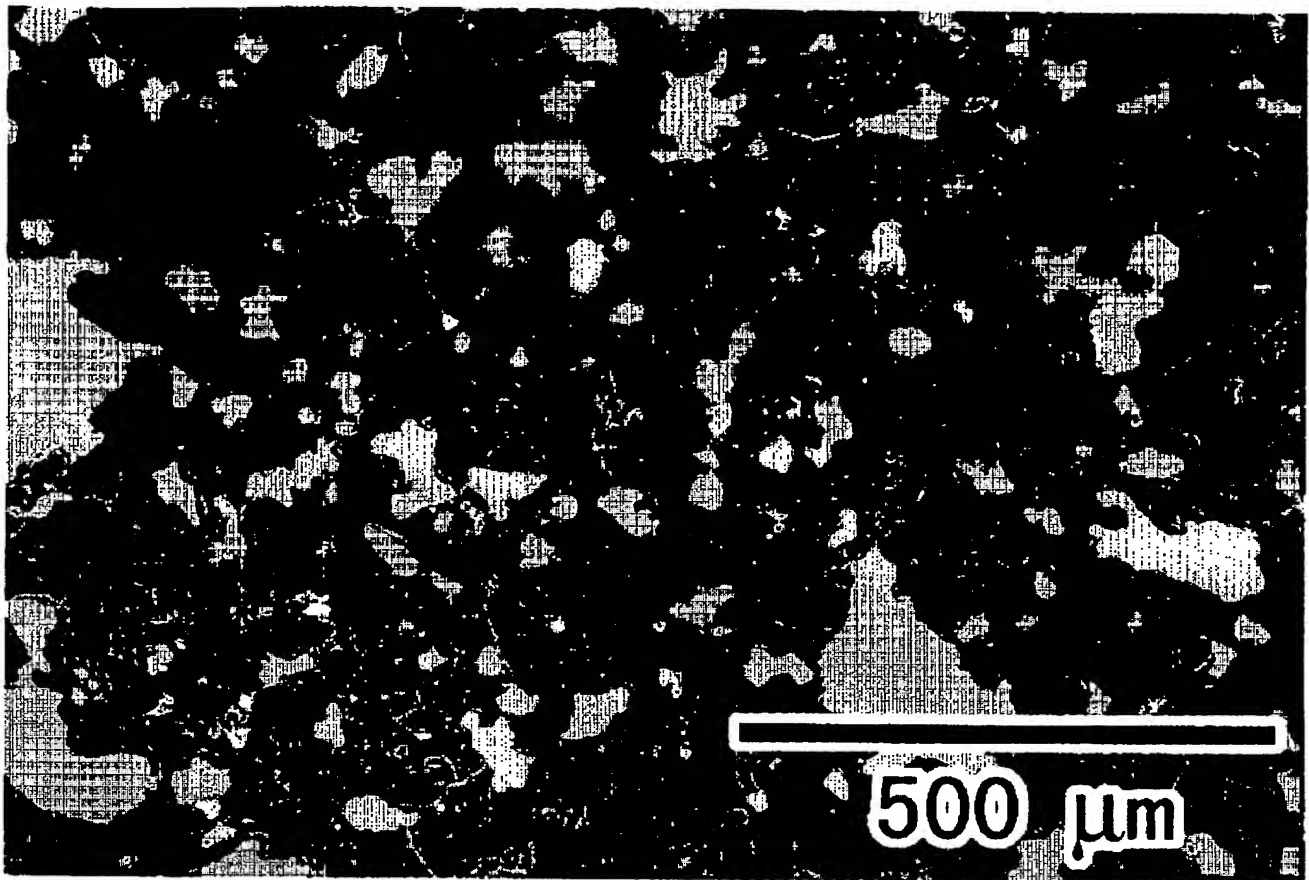
[Drawing 3]



[Drawing 6]



[Drawing 4]



---

[Translation done.]

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☒ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☒ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**